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Web Page URLs for STN Seminar Schedule - N. America
NEWS
NEWS
     2
                 "Ask CAS" for self-help around the clock
                CA/CAPLUS - Russian Agency for Patents and Trademarks
NEWS
        FEB 25
                 (ROSPATENT) added to list of core patent offices covered
NEWS 4
        FEB 28
                PATDPAFULL - New display fields provide for legal status
                data from INPADOC
NEWS 5 FEB 28
                BABS - Current-awareness alerts (SDIs) available
                MEDLINE/LMEDLINE reloaded
NEWS 6 FEB 28
NEWS 7 MAR 02
                GBFULL: New full-text patent database on STN
NEWS 8 MAR 03
                REGISTRY/ZREGISTRY - Sequence annotations enhanced
                MEDLINE file segment of TOXCENTER reloaded
NEWS 9 MAR 03
NEWS 10 MAR 22
                KOREAPAT now updated monthly; patent information enhanced
NEWS 11 MAR 22
                Original IDE display format returns to REGISTRY/ZREGISTRY
NEWS 12 MAR 22
                PATDPASPC - New patent database available
NEWS 13 MAR 22
                REGISTRY/ZREGISTRY enhanced with experimental property tags
NEWS 14 APR 04
                EPFULL enhanced with additional patent information and new
                fields
NEWS 15 APR 04
                EMBASE - Database reloaded and enhanced
NEWS 16 APR 18
                New CAS Information Use Policies available online
NEWS 17 APR 25
                Patent searching, including current-awareness alerts (SDIs),
                based on application date in CA/CAplus and USPATFULL/USPAT2
                may be affected by a change in filing date for U.S.
                applications.
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NEWS EXPRESS JANUARY 10 CURRENT WINDOWS VERSION IS V7.01a, CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP), AND CURRENT DISCOVER FILE IS DATED 10 JANUARY 2005

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FILE 'HOME' ENTERED AT 09:48:14 ON 27 APR 2005

=> file reg COST IN U.S. DOLLARS FULL ESTIMATED COST ENTRY SESSION 0.21 0.21

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STRUCTURE FILE UPDATES: 26 APR 2005 HIGHEST RN 849322-79-8 DICTIONARY FILE UPDATES: 26 APR 2005 HIGHEST RN 849322-79-8

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=> logoff hold COST IN U.S. DOLLARS

FULL ESTIMATED COST

SINCE FILE TOTAL ENTRY SESSION 0.43 0.64

SESSION WILL BE HELD FOR 60 MINUTES
STN INTERNATIONAL SESSION SUSPENDED AT 09:48:28 ON 27 APR 2005

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NEWS 2 "Ask CAS" for self-help around the clock

NEWS 3 FEB 25 CA/CAPLUS - Russian Agency for Patents and Trademarks (ROSPATENT) added to list of core patent offices covered

NEWS 4 FEB 28 PATDPAFULL - New display fields provide for legal status

data from INPADOC

NEWS 5 FEB 28 BABS - Current-awareness alerts (SDIs) available

NEWS 6 FEB 28 MEDLINE/LMEDLINE reloaded

NEWS 7 MAR 02 GBFULL: New full-text patent database on STN

NEWS 8 MAR 03 REGISTRY/ZREGISTRY - Sequence annotations enhanced

NEWS 9 MAR 03 MEDLINE file segment of TOXCENTER reloaded

NEWS 10 MAR 22 KOREAPAT now updated monthly; patent information enhanced

NEWS 11 MAR 22 Original IDE display format returns to REGISTRY/ZREGISTRY

NEWS 12 MAR 22 PATDPASPC - New patent database available

NEWS 13 MAR 22 REGISTRY/ZREGISTRY enhanced with experimental property tags

NEWS 14 APR 04 EPFULL enhanced with additional patent information and new fields

NEWS 15 APR 04 EMBASE - Database reloaded and enhanced

NEWS 16 APR 18 New CAS Information Use Policies available online

NEWS 17 APR 25 Patent searching, including current-awareness alerts (SDIs), based on application date in CA/CAplus and USPATFULL/USPAT2 may be affected by a change in filing date for U.S. applications.

NEWS EXPRESS JANUARY 10 CURRENT WINDOWS VERSION IS V7.01a, CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP), AND CURRENT DISCOVER FILE IS DATED 10 JANUARY 2005

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FILE 'HOME' ENTERED AT 13:26:02 ON 27 APR 2005

=> e reg

THIS COMMAND NOT AVAILABLE IN THE CURRENT FILE The EXPAND command is used to look at the index in a file which has an index. This file does not have an index.

=> file reg

COST IN U.S. DOLLARS

SINCE FILE TOTAL ENTRY SESSION

0.21

FULL ESTIMATED COST 0.21

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New CAS Information Use Policies, enter HELP USAGETERMS for details.

TSCA INFORMATION NOW CURRENT THROUGH JANUARY 18, 2005

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************* The CA roles and document type information have been removed from * the IDE default display format and the ED field has been added, * effective March 20, 2005. A new display format, IDERL, is now available and contains the CA role and document type information. *****************

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Experimental and calculated property data are now available. For more information enter HELP PROP at an arrow prompt in the file or refer to the file summary sheet on the web at:

http://www.cas.org/ONLINE/DBSS/registryss.html

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chain nodes :

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 chain bonds :

1-2 1-14 1-15 1-16 2-3 2-17 2-18 3-4 3-19 3-20 4-5 5-6 5-21 5-33 6-7 6-22 6-23 7-8 8-9 8-40 9-10 9-24 9-32 10-11 11-12 11-25 11-26 12-13 12-27 12-28 13-29 13-30 13-31 32-37 32-38 32-39 33-34 33-35 33-36 exact/norm bonds :

3-4 4-5 6-7 7-8 8-40 9-10 10-11

exact bonds :

1-2 1-14 1-15 1-16 2-3 2-17 2-18 3-19 3-20 5-6 5-21 5-33 6-22 6-23 8-9 9-24 9-32 11-12 11-25 11-26 12-13 12-27 12-28 13-29 13-30 13-31 32-37 32-38 32-39 33-34 33-35 33-36

Match level :

1:CLASS 2:CLASS 3:CLASS 4:CLASS 5:CLASS 6:CLASS 7:CLASS 8:CLASS 9:CLASS 10:CLASS 11:CLASS 12:CLASS 13:CLASS 14:CLASS 15:CLASS 16:CLASS 17:CLASS 19:CLASS 20:CLASS 21:CLASS 22:CLASS 23:CLASS 24:CLASS 18:CLASS 25:CLASS 27:CLASS 28:CLASS 29:CLASS 30:CLASS 31:CLASS 32:CLASS 33:CLASS 26:CLASS 34:CLASS 35:CLASS 36:CLASS 37:CLASS 38:CLASS 39:CLASS 40:CLASS

STRUCTURE UPLOADED L1

=> d 11

L1 HAS NO ANSWERS

STR L1

Structure attributes must be viewed using STN Express query preparation.

=> search l1 exact full

FULL SEARCH INITIATED 13:26:58 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 149 TO ITERATE

100.0% PROCESSED 149 ITERATIONS

1 ANSWERS

SEARCH TIME: 00.00.01

L2 1 SEA EXA FUL L1

=> d scan

L2 1 ANSWERS REGISTRY COPYRIGHT 2005 ACS on STN

MF C12 F24 O4

PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

ALL ANSWERS HAVE BEEN SCANNED

=> fikle caplus

0 FIKLE

0 CAPLUS

L3 0 FIKLE CAPLUS

(FIKLE (W) CAPLUS)

=> file caplus

COST IN U.S. DOLLARS

SINCE FILE TOTAL ENTRY SESSION 64.31 64.52

FULL ESTIMATED COST

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FILE COVERS 1907 - 27 Apr 2005 VOL 142 ISS 18 FILE LAST UPDATED: 26 Apr 2005 (20050426/ED)

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This file contains CAS Registry Numbers for easy and accurate substance identification.

=> 13

L4 0 L3

=> 12

L5 12 L2

=> d 15 1-12 ti

- L5 ANSWER 1 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Method for producing fluorinated compound and fluorination apparatus
- L5 ANSWER 2 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Preparation of compounds while analyzing products using near-IR spectroscopy
- L5 ANSWER 3 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Processes for producing fluorinated esters, fluorinated acyl fluorides, and fluorinated vinyl ethers
- L5 ANSWER 4 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Process for producing product of decomposition of fluorinated ester compound
- L5 ANSWER 5 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Process for preparing fluorinated esters using gas chromatography
- L5 ANSWER 6 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI A new route to perfluoro(alkoxyalkanoyl) fluorides from non-fluorinated compounds
- L5 ANSWER 7 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Process for producing fluorinated ester compounds
- L5 ANSWER 8 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Manufacture of perfluoro compounds and derivatives
- L5 ANSWER 9 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Process for the preparation of etheric oxygen-containing perfluoroacyl fluorides and perfluorovinyl ethers
- L5 ANSWER 10 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI A new route to perfluorinated vinyl ether monomers: synthesis of perfluoro(alkoxyalkanoyl) fluorides from non-fluorinated compounds
- L5 ANSWER 11 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Synthesis of versatile poly- and perfluorinated compounds by utilizing

direct fluorination, a new route to perfluoro(propyl vinyl ether) monomer: synthesis of perfluoro(2-propoxypropionyl) fluoride from non-fluorinated compounds

- L5 ANSWER 12 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Process for producing fluorine compound through liquid-phase fluorination

=> d 15 1-12 ti fbib abs

- L5 ANSWER 1 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Method for producing fluorinated compound and fluorination apparatus
- AN 2004:354898 CAPLUS
- DN 140:356951
- TI Method for producing fluorinated compound and fluorination apparatus
- IN Suzuki, Yasuhiro; Watanabe, Kunio; Yanase, Koichi
- PA Asahi Glass Company, Limited, Japan
- SO PCT Int. Appl., 35 pp.
- CODEN: PIXXD2
- DT Patent
- LA Japanese
- FAN.CNT 1

	1111110111 1																	
	PATENT NO.					KIND DATE			APPLICATION NO.						DATE			
ΡI	WO 2004035518			A1 2004042			0429	,	WO 2	003-	JP13	20031017						
		W:	ΑE,	AG,	AL,	AM,	ΑT,	AU,	ΑZ,	BA,	BB,	BG,	BR,	BY,	ΒZ,	CA,	CH,	CN,
			CO,	CR,	CU,	CZ,	DE,	DK,	DM,	DΖ,	EC,	EE,	EG,	ES,	FI,	GB,	GD,	GE,
			GH,	GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	KΕ,	KG,	KR,	KZ,	LC,	LK,	LR,
			LS,	LT,	LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	ΜZ,	NI,	NO,	NZ,	OM,
			PG,	PH,	PL,	PT,	RO,	RU,	SC,	SD,	SE,	SG,	SK,	SL,	SY,	ТJ,	TM,	TN,
			TR,	TT,	TZ,	UA,	ŪĠ,	US,	UΖ,	VC,	VN,	ΥU,	ZA,	ZM,	zw			
		RW:	GH,	GM,	ΚE,	LS,	MW,	MZ,	SD,	SL,	SZ,	TZ,	ŪĠ,	ZM,	ZW,	AM,	ΑZ,	BY,
			KG,	ΚZ,	MD,	RU,	ΤJ,	TM,	ΑT,	ΒE,	BG,	CH,	CY,	CZ,	DE,	DK,	EE,	ES,
			FI,	FR,	GB,	GR,	HU,	ΙE,	IT,	LU,	MC,	NL,	PT,	RO,	SE,	SI,	SK,	TR,
			BF,	ВJ,	CF,	CG,	CI,	CM,	GA,	GN,	GQ,	GW,	ML,	MR,	ΝE,	SN,	TD,	TG
											JP 2	002-	3046	A 20021018				

OS CASREACT 140:356951

AB Disclosed is a method for producing a fluorinated compound wherein a section for introducing fluorine, a section for introducing a raw material compound to be fluorinated, a fluorination region and a section for discharging a fluorinated compound are connected with a circulating circuit in which an inert liquid is flowing in one direction, and fluorination is effected by introducing the raw material compound and fluorine into an inert liquid in said circuit, characterized in that fluorine is introduced into the flow of an inert liquid containing substantially no raw material compound The method

allows the continuous production of a fluorinated compound with improved efficiency, with the suppression of an undesired side reaction. Thus, a mixture of F(g) (78 g/h) and N (57 g/h) and CF3CF2CF2OCF(CF3)CO2CH2CH(CH3)OC H2CH2CH3 (I) (52 g/h) were continuously fed into a inert liquid of CF3CF2CF2OCF(CF3)CF2OCF(CF3)COF (4 kg) circulating through a stainless steel tube at 20° for 300 h in an apparatus described above to give a reaction mixture containing 95.2 mol% CF3CF2CF2OCF(CF3)CO2CF2(CF3)OCF2CF2CF3 (product), 3.0 mol% partially fluorinated I, and 1.8% mol% fluorinated byproducts derived from cleavage of carbon bonds.

RE.CNT 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

- L5 ANSWER 2 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Preparation of compounds while analyzing products using near-IR spectroscopy
- AN 2002:802409 CAPLUS
- DN 137:325157
- TI Preparation of compounds while analyzing products using near-IR spectroscopy

```
Yamamoto, Kiyoshi; Kakita, Reiko; Okamoto, Shuichi
IN
PA
     Japan Carlit Co., Ltd., Japan
so
     Jpn. Kokai Tokkyo Koho, 6 pp.
     CODEN: JKXXAF
     Patent
DT
LΑ
     Japanese
FAN.CNT 1
                                        APPLICATION NO.
     PATENT NO.
                      KIND
                               DATE
                                                                 DATE
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PΙ
     JP 2002308827
                         A2
                               20021023
                                          JP 2001-108764
                                                                 20010406
                                           JP 2001-108764
                                                                 20010406
     Compds. are prepared by chemical conversion of H atom of compds. showing H-X (X
AB
     = arbitrary atom) vibrational absorption in near-IR region while
     monitoring amount of H atom by near-IR absorption. Preparation of
     perfluoro[1,2-bis(2-ethylhexyloxycarbonyl)]hexane by fluorination of
     bis(2-ethylhexyl) phthalate was carried out while monitoring the reactant
     conversion by near-IR spectrum.
     ANSWER 3 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
L5
TI
     Processes for producing fluorinated esters, fluorinated acyl fluorides,
     and fluorinated vinyl ethers
     2002:539635 CAPLUS
AN
DN
     137:94172
     Processes for producing fluorinated esters, fluorinated acyl fluorides,
ΤI
     and fluorinated vinyl ethers
IN
     Okazoe, Takashi; Watanabe, Kunio; Tatematsu, Shin; Murofushi, Hidenobu;
     Sato, Masakuni; Ito, Masahiro; Yanase, Koichi; Suzuki, Yasuhiro
PA
     Asahi Glass Company, Limited, Japan
SO
     PCT Int. Appl., 39 pp.
     CODEN: PIXXD2
DT
     Patent
TιA
     Japanese
FAN.CNT 1
     PATENT NO.
                        KIND
                              DATE
                                         APPLICATION NO.
                                                               DATE
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                               _____
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                        A1
                               20020718 WO 2002-JP236
PΙ
     WO 2002055471
                                                                20020116
        W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
            CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
            GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LK, LR, LS,
            LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL,
            PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA,
            UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
        RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH,
            CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR,
            BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
                                          JP 2001-8252 A 20010116
                              20031015
                                          EP 2002-715755
    EP 1352892
                         A1
                                                                 20020116
        R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
            IE, SI, LT, LV, FI, RO, MK, CY, AL, TR
                                          JP 2001-8252
                                                              A 20010116
                                                              W 20020116
                                           WO 2002-JP236
     ZA 2003005170
                         Α
                               20040709
                                           ZA 2003-5170
                                                                 20020116
                                                             A 20010116
                                          JP 2001-8252
os
    MARPAT 137:94172
    Fluorinated esters are prepared in shorter steps including a
AB
     transesterification step in which RACH2OH (RA = monovalent organic group) and
     RAFCOOCF2RAF [RAF = (fluorinated) RA] are subjected to transesterification
     to obtain RAFCOOCH2RA, and a fluorination step in which the compound
     obtained is fluorinated in a liquid phase to obtain a reaction product
```

RE.CNT 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

fluorinated esters.

containing RAFCOOCF2RAF. Fluorinated acyl fluorides and perfluorovinyl ethers (e.g., CF3CF2CF2OCF:CF2) can be prepared in large scale with low cost from

ANSWER 4 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN L5 TI Process for producing product of decomposition of fluorinated ester compound 2002:465955 CAPLUS AN 137:47203 DN Process for producing product of decomposition of fluorinated ester ΤI IN Watanabe, Kunio; Suzuki, Yasuhiro; Yanase, Koichi; Okazoe, Takashi PA Asahi Glass Company, Limited, Japan SO PCT Int. Appl., 33 pp. CODEN: PIXXD2 DT Patent Japanese LA FAN.CNT 1 PATENT NO. KIND APPLICATION NO. DATE DATE _ _ _ _ -----20020620 WO 2001-JP10889 ΡI WO 2002048085 A1 20011212 AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG JP 2000-379101 A 20001213 AU 2002021128 **A5** 20020624 AU 2002-21128 20011212 JP 2000-379101 20001213 WO 2001-JP10889 20011212 os CASREACT 137:47203; MARPAT 137:47203 GI

AΒ

Disclosed is a process for production of fluorinated ketones of formula

RAFRBFCO (RAF = F, monovalent organic group; RBF = monovalent organic group) and/or fluorinated acyl fluoride of formula RCFCOF (RCF = monovalent organic group) with high productivity which comprises efficiently decomposing a fluorinated ester compound of formula RCFCO2CFRAFRBF (RAF, RBF, RCF = same as above) at a low temperature and a high reaction rate. In this process, a fluorinated ester compound having a decomposable ester bond is decomposed at the ester bond to obtain a decomposition product. The ester bond decomposition reaction is conducted in the presence of KF at a temperature of 200° or lower substantially without using a solvent. The reaction is conducted while continuously supplying the fluorinated ester compound to the reaction zone and continuously discharging the decomposition products from the reaction Thus, a 59:41 mixture of 5-hydroxy-1,3-dioxane and 1,3-dioxolane-4-methanol (100 g) and 10.7 g Et3N were charged into a flask, and stirred at ≤10°, followed by adding dropwise 351.0 g FCOCF(CF3)OCF2CF2CF3 at ≤10° over a period of 400 min, and the resulting mixture was stirred at room temperature for 1 h and

treated

with 500 mL H2O at ≤15° and then with 1,000 mL dichloropentafluoropropane (AK225). The bottom layer was separated, washed twice with 500 mL H2O, dried over MgSO4, filtered, concentrated by an evaporator, and vacuum-distilled at 59-62° and 0.4 kPa to give a mixture of esters (I and II) (328.0 g). Diluted F (20 volume % in N) was blown at 17.04 L/h and 25° into 1,701 g R-113 for 1 h in an autoclave connected at the gas outlet to a condenser (kept at 20°), a NaF pellet-packed layer, and a condenser (kept at -10°) in series, followed by injecting a solution of a mixture of I and II esters (115 g) in 863 q R-113 over a period of 24.8 h while blowing diluted F into the reaction solution A solution of benzene in R-113 (0.04 g/mL, 30 mL) was injected into the reaction mixture at 0.15 MPa and 25-40° while blowing diluted F into the reaction solution and then stirred for 0.3 h. A solution of benzene

R-113 (20 mL) was again injected into the reaction mixture at 0.15 MPa and 40° while blowing diluted F into the reaction solution and then stirred for 0.3 h. After repeating the same procedure one more time upon which a total of 3.14 g benzene and 70 mL R-113 was added, the reaction mixture was stirred for 1.0 h to give a crude reaction liquid containing perfluorinated esters (III) (68% yield) and (IV) (93% yield). The crude reaction liquid (135.1 q) and 2.98 q KF were charged into a reactor and vigorously stirred at 91° for 5 h. A liquid sample (118.8 g) was collected at the outlet of the reflux condenser, which contained CF3CF(OCF2CF2CF3)COF as the main product and perfluoro-1,3-dioxan-5-one (V) (83% yield) and perfluoro-1,3-dioxolane-4-carbonyl fluoride (VI) (62% yield).

RE.CNT 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

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ANSWER 5 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
L5
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Process for preparing fluorinated esters using gas chromatography ΤI

2002:391677 CAPLUS AN

DN 136:401447

Process for preparing fluorinated esters using gas chromatography ΤI

Kawahara, Kengo; Isemura, Tsuguhide; Okazoe, Takashi ΙN

Asahi Glass Company, Limited, Japan PΑ

PCT Int. Appl., 19 pp. SO

CODEN: PIXXD2

DTPatent

LΑ Japanese

FAN.CNT 1

in

	PATENT NO.					KIND DATE			APPLICATION NO.						DATE				
							-												
ΡI	WO 2002040437				A1 20020523				WO 2001-JP10116						20011120				
		W:	ΑE,	AG,	AL,	AM,	AT,	ΑU,	AZ,	BA,	BB,	BG,	BR,	BY,	ΒZ,	CA,	CH,	CN,	
			CO,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	EE,	ES,	FI,	GB,	GD,	GE,	GH,	
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        BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
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                                       WO 2001-JP10116
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       AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
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                                       JP 2000-353340
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                                       WO 2001-JP10116
                                                           A1 20011120
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OS CASREACT 136:401447

AB A process for determining accurately the amount of a fluorinated ester in the preparation thereof or determining accurately the consumption of a fluorinated ester

and the amount of a formed substance in the chemical transformation of the ester allows the progress of the degree of fluorination and thermal decomposition to be monitored and enables efficient preparation of the objective

substance in a yield equal to or higher than an objective level. That is, a process for preparing a fluorinated ester from starting compds. by a chemical reaction is characterized in that the reaction is continued until the yield of the ester reaches a predetd. level as determined by gas chromatog. with a nonpolar column. This process is also useful for quality control in a manufacturing process using fluorinated ester. Thus, NaF was added to 1,1,2-trichlorotrifluoroethane (R-113) in a autoclave and cooled to -10°, to which N was blown into the mixture for 1 h and then 20% F in N for 1 h, followed by injecting a solution of MeCH2CH2OCHMeCH2O2CCF(CF3)O(CF 2)3F in R-113 over 19.4 h. A solution of benzene in R-113 was injected to the reaction mixture while blowing 25%F/N into the mixture and the outlet valve of the autoclave was closed and then the inlet valve of the autoclave was closed when the pressure reached at 0.12 MPa, followed by stirring the resulting mixture for 1 h. The above procedure (benzene treatment) was repeated four times while the temperature rose from -10° to room temperature and five times at room temperature, followed by blowing N into the

resulting mixture for 1 h. A anal. sample was taken by decantation and analyzed by GC at 270° using a nonpolar capillary column (J & W Inc., DB-1, 60 m length, inner diameter 0.25 mm, membrane thickness 1.0 µm) (main column) and an inactivated fused silica hollow capillary column (GL Science Inc., length 1 m, inner diameter 0.530 mm, and outer diameter

0.660 mm) (precolumn) which were connected by a capillary column connector. A hydrogen flame ionization detector was used at 280° and the structure of each peak was determined by mass spectrometer. The anal confirmed that two peaks corresponding to perfluorinated ester, i.e. CF3CF2CF2CCF(CF3)CF2CCF(CF3)OCF2CF2CF3, and perfluoroacyl fluoride, i.e. CF3CF2CF2CCF(CF3)COF, were formed in 95 and 0.6%, resp., and completely separated in the gas chromatogram. The perfluorinated ester was analyzed substantially without decomposition

RE.CNT 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

- TI A new route to perfluoro(alkoxyalkanoyl) fluorides from non-fluorinated compounds
- AN 2002:285166 CAPLUS
- DN 137:294705
- TI A new route to perfluoro(alkoxyalkanoyl) fluorides from non-fluorinated compounds
- AU Okazoe, Takashi; Watanabe, Kunio; Itoh, Masahiro; Shirakawa, Daisuke; Murofushi, Hidenobu; Okamoto, Hidekazu; Kawahara, Kengo; Tatematsu, Shin
- CS Res. Cent., Asahi Glass Co., Ltd., Yokohama, 221-8755, Japan
- SO Asahi Garasu Kenkyu Hokoku (2001), 51, 33-37 CODEN: AGKHAD; ISSN: 0004-4210
- PB Asahi Garasu K.K. Chuo Kenkyusho
- DT Journal
- LA Japanese
- OS CASREACT 137:294705
- A new synthetic procedure for the preparation of perfluoro(alkoxyalkanoyl) AB fluorides, which are precursors to perfluorinated vinyl ether monomers, from non-fluorinated alkoxyalcs. has been developed. The new procedure involves esterification of non-fluorinated alkoxyalcs. with perfluoro(alkoxyalkanoyl) fluorides, direct fluorination of the resulting esters, i.e. alkoxyalcs. perfluoro(alkoxyalkanoyl) esters, by fluorine gas, and thermal decomposition of perfluorinated esters into perfluoro(alkoxyalkanoyl) fluorides in the presence of NaF as the catalyst. Available perfluoro (alkoxyalkanoyl) fluorides such as perfluoro(2-propoxypropionyl) fluoride, so called hexafluoropropylene oxide (HFPO) dimer, can be multiplied by the use of the hydrocarbon counterpart alcs. and fluorine gas as raw materials. In the case that the desired perfluoro (alkoxyalkanoyl) fluoride is not readily available, it can be obtained from its hydrocarbon counterpart alc. and an available perfluoroacyl fluoride. Thus, 1-propoxy-1-propanol was esterified with perfluoro(2-propoxypropionyl) fluoride (I) at 25-35° to give 99.2% 2-propoxy-Pr perfluoro(2-propoxypropionate) which was fluorinated by 20% F(g) in N in 1,1,2-trichlorotrifluoroethane at room temperature for 63.7 h followed by injecting a solution of benzene in 1,1,2-trichlorotrifluoroethane after closing the F(g) inlet valve, and the resulting mixture was allowed to react at 40° to give 93.2% perfluoro(2-propoxypropyl 2-propoxypropionate) (II). A suspension of NaF in II was heated at 140° for 15 in an oil bath with vigorous stirring to give 94.2% I.
- L5 ANSWER 7 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Process for producing fluorinated ester compounds
- AN 2002:256214 CAPLUS
- DN 136:294541
- TI Process for producing fluorinated ester compounds
- IN Okazoe, Takashi; Watanabe, Kunio; Tatematsu, Shin; Yanase, Koichi; Suzuki,
 Yasuhiro; Shirakawa, Daisuke
- PA Asahi Glass Co., Ltd., Japan
- SO PCT Int. Appl., 52 pp.
- CODEN: PIXXD2
- DT Patent LA Japanese
- FAN.CNT 1

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ΡI	WO 2002026688			A 1		20020404		WO 2001-JP8433					2001:0927					
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                                           JP 2000-295141
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                                                              A1 20010927
     CASREACT 136:294541; MARPAT 136:294541
     This document discloses a process for producing an industrially useful
     fluorinated ester compound and a fluorine compound such as an acid fluoride
     compound The title compds. are intermediates for monomers. The process for
     producing a fluorinated ester compound comprises fluorinating in a liquid
     phase a compound which is an ester of a hydroxyl compound with a compound
having
     an acyl fluoride group and which has a structure capable of being
     fluorinated, and said process is characterized in that the ester compound
     being fluorinated is in the form of a liquid mixture with the compound having
     acyl fluoride group.
             THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE.CNT 5
             ALL CITATIONS AVAILABLE IN THE RE FORMAT
     ANSWER 8 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
    Manufacture of perfluoro compounds and derivatives
     2002:256213 CAPLUS
     136:294528
    Manufacture of perfluoro compounds and derivatives
    Watanabe, Kunio; Okazoe, Takashi; Tatematsu, Shin; Shirakawa, Daisuke
    Asahi Glass Co., Ltd., Japan
     PCT Int. Appl., 42 pp.
    CODEN: PIXXD2
     Patent
     Japanese
FAN.CNT 1
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                                                                 DATE
    PATENT NO.
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                        A1 20020404 WO 2001-JP8367
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            UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
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                         Α5
                                                                  20010926
                                                               A 20000927
                                           JP 2000-295134
                                                               W 20010926
                                           WO 2001-JP8367
    CASREACT 136:294528; MARPAT 136:294528
    Perfluoro compds. and perfluoro acid fluorides are efficiently prepared
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AB Compds. of the general formula RAFOCF(CF3)CF2OCOCF(CF3)ORBF are prepared by fluorinating a hydrogen-containing fluorinated compound, e.g., RAHOCH(CH3)CH2OCOCF(CF3)ORBH in a liquid phase in the presence of a product of partial fluorination of the hydrogen-containing fluorinated compound, e.g., RAFOCH(CF3)CF2OCOCF(CF3)ORBF or RAFOCF(CF3)CHFOCOCF(CF3)ORBF (RAH, RBH = monovalent organic group; RAF, RBF = monovalent perfluorinated organic group). Further, the compds. thus prepared can be converted into perfluoro acid fluorides via ester linkage cleavage, and the perfluoro acid fluorides can be further converted into perfluoro vinyl ethers via pyrolysis.

RE.CNT 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

- L5 ANSWER 9 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Process for the preparation of etheric oxygen-containing perfluoroacyl fluorides and perfluorovinyl ethers
- AN 2002:107288 CAPLUS
- DN 136:151577
- TI Process for the preparation of etheric oxygen-containing perfluoroacyl fluorides and perfluorovinyl ethers
- IN Okazoe, Takashi; Murofushi, Hidenobu; Watanabe, Kunio; Tatematsu, Shin
- PA Asahi Glass Company, Limited, Japan
- SO PCT Int. Appl., 42 pp.
 - CODEN: PIXXD2
- DT Patent
- LA Japanese
- FAN.CNT 1

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			LT,	LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NO,	NZ,	PL,	PT,	RO,
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			ВJ,	CF,	CG,	CI,	CM,	GΑ,	GN,	GQ,	GW,	ML,	MR,	NE,	SN,	TD,	TG	
	, , ,										TP 2	000-	2211	57		2 2	nnnn	731

- OS MARPAT 136:151577
- AB The invention relates to a process for the preparation of industrially useful fluorine compds. such as acyl fluorides, i.e., a process which comprises reacting a compound of the following general formula: RAHOCH(CH3)CH2OCOCF(CF3)ORBf having a fluorine content of 30% by mass or above with fluorine in a liquid phase to thereby obtain a compound of the following general formula: RAfOCF(CF3)CF2OCOCF(CF3)ORBf, and subjecting this compound to ester linkage cleavage (wherein RAH is C1-20 alkyl or C1-20 alkyl containing one or more etheric oxygen atoms; RAf is a group derived from RAH by perfluorination; and RBf is C1-20 perfluoroalkyl or C1-20 perfluoroalkyl containing one or more etheric oxygen atoms). Thus, CH3CH2CH2OCH(CH3)CH2OH obtained from propylene oxide and 1-propanol were reacted with CF3CF2CF2OCF(CF3)COF to give CF3CF2CF2OCF(CF3)COOCH2CH(CH3)OC H2CH2CH3, fluorinated with F2 in R 113 containing NaF to give CF3CF2CF2OCF(CF3)COOCF2CF(CF3)OCF2CF2CF3, thermally decomposed to give CF3CF2CF2OCF(CF3)COF, then give CF3CF2CF2OCF:CF2.
- RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT
- L5 ANSWER 10 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI A new route to perfluorinated vinyl ether monomers: synthesis of perfluoro(alkoxyalkanoyl) fluorides from non-fluorinated compounds
- AN 2001:872315 CAPLUS
- DN 136:135058
- TI A new route to perfluorinated vinyl ether monomers: synthesis of perfluoro(alkoxyalkanoyl) fluorides from non-fluorinated compounds
- AU Okazoe, Takashi; Watanabe, Kunio; Itoh, Masahiro; Shirakawa, Daisuke; Tatematsu, Shin
- CS Research Center, Asahi Glass Co. Ltd., Kanagawa-ku, Yokohama, 221-8755, Japan

- SO Journal of Fluorine Chemistry (2001), 112(1), 109-116 CODEN: JFLCAR; ISSN: 0022-1139
- PB Elsevier Science S.A.
- DT Journal
- LA English
- An ew synthetic procedure for the preparation of various perfluoro(alkoxyalkanoyl) fluorides, which are precursors to perfluorinated vinyl ether monomers, from non-fluorinated alkoxy alcs. was developed. Available perfluoro(alkoxyalkanoyl) fluorides such as perfluoro(2-propoxypropionyl) fluoride, so-called HFPO dimer, can be multiplied by the use of the hydrocarbon counterpart alcs. and fluorine gas as raw materials. In the case that the desired perfluoro(alkoxyalkanoyl) fluoride is not readily available, it can be obtained from its hydrocarbon counterpart alc. and an available perfluoroacyl fluoride.
- RE.CNT 30 THERE ARE 30 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT
- L5 ANSWER 11 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Synthesis of versatile poly- and perfluorinated compounds by utilizing direct fluorination, a new route to perfluoro(propyl vinyl ether) monomer: synthesis of perfluoro(2-propoxypropionyl) fluoride from non-fluorinated compounds
- AN 2001:186960 CAPLUS
- DN 134:367222
- TI Synthesis of versatile poly- and perfluorinated compounds by utilizing direct fluorination, a new route to perfluoro(propyl vinyl ether) monomer: synthesis of perfluoro(2-propoxypropionyl) fluoride from non-fluorinated compounds
- AU Okazoe, Takashi; Watanabe, Kunio; Itoh, Masahiro; Shirakawa, Daisuke; Murofushi, Hidenobu; Okamoto, Hidekazu; Tatematsu, Shin
- CS Research Center, Asahi Glass Co., Ltd., Yokohama, 221-8755, Japan
- SO Advanced Synthesis & Catalysis (2001), 343(2), 215-219 CODEN: ASCAF7; ISSN: 1615-4150
- PB Wiley-VCH Verlag GmbH
- DT Journal
- LA English
- AB Perfluoro(2-propoxypropionyl) fluoride, which is the precursor of the perfluorinated Pr vinyl ether (PPVE) monomer of an industrially important perfluoroalkoxy copolymer (PFA), was synthesized by utilizing direct fluorination of the non-fluorinated counterpart for the first time. The partially-fluorinated ester synthesized from the desired perfluorinated acid fluoride itself and the non-fluorinated alc., which has a carbon skeleton corresponding to the desired compound, was perfluorinated by liquid-phase direct fluorination with elemental fluorine. Degradation of the resulting perfluorinated ester gave 2 mols. of the desired acid fluoride. In a sense, this process can be called self-multiplication of a perfluorinated acid fluoride from a non-fluorinated alc.
- RE.CNT 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT
- L5 ANSWER 12 OF 12 CAPLUS COPYRIGHT 2005 ACS on STN
- TI Process for producing fluorine compound through liquid-phase fluorination
- AN 2000:688202 CAPLUS
- DN 133:266514
- TI Process for producing fluorine compound through liquid-phase fluorination
- IN Okazoe, Takashi; Watanabe, Kunio; Tatemasu, Shin; Murofushi, Hidenobu
- PA Asahi Glass Company, Limited, Japan
- SO PCT Int. Appl., 83 pp. CODEN: PIXXD2
- DT Patent
- LA Japanese
- FAN.CNT 2

PATENT NO. KIND DATE APPLICATION NO. DATE

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CASREACT 133:266514; MARPAT 133:266514 OS

A process for producing a fluorine compound such as industrially useful AΒ perfluorinated alkanoic acid fluoride derivative, from inexpensive materials in short steps and high yields is described. The process comprises reacting a compound RA-E1, e.g., RACH2OH, with a compound RB-E2, e.g., XCORB, to form a compound RA-E-RB (I), e.g., RACH2OCORB, fluorinating the compound I in a liquid phase to form a compound RAF-EF-RBF (II), e.g., RAFCF2OCORBF, and converting the compound II into a compound RAF-EF1, e.g., RAFCOF, and/or a compound RBF-EF2, e.g., RBFCOF, [wherein RA, RB = monovalent saturated (halo) hydrocarbyl optionally containing heteroatoms, monovalent organic group which can be converted into RHF group in the liquid phase; RHF = group derived by replacing ≥1 H atom(s) of monovalent (partially halogenated) saturated hydrocarbyl optionally containing heteroatom(s) with fluorine atom(s); RAF and RBF are fluorinated RA and RB, resp.; X = halo]. Thus, 46.5 g CF3 (CF3CF2CF2O) CFCOF was added dropwise to 16.5 g CH3(CH3CH2CH2O)CHCH2OH at 26-31° over a period of 2 h with bubbling N into the reaction mixture to give, after workup and vacuum distillation, CF3CF(OCF2CF2CF3)CO2CH2CH(OCH2CH3)CH3 (III) in three fractions 29 g (68% purity), 19 g (98% purity), and 4 g (97 % purity). The last two fractions were combined, a portion of which (19.5 g) was dissolved in 250 g R-113 to give a solution of III. The latter solution was injected into a cooled (-10°) mixture of 324 g R-113 and 26.1 g NaF in an autoclave over a period of 17.4 h while bubbling F(g) at 5.66 L/h in to the reaction mixture to give CF3CF(OCF2CF2CF3)CO2CF2CF(OCF2CF2CF3)CF3 in 68% based on 19NMR which was purified by vacuum distillation The latter compound (15 g) was placed in a stainless steel ampule and heated at 200° for 2 h to give CF3CF(OCF2CF2CF3)COF in 85% yield based on 19NMR.

THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD RE.CNT 6 ALL CITATIONS AVAILABLE IN THE RE FORMAT

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DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS) SINCE	FILE TOTAL
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